ESTCP
Cost and Performance Report

(ER-200545)

Integrated Ion Exchange Regeneration Process for Perchlorate in Drinking Water

August 2010

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ACRONYMS AND ABBREVIATIONS

bgs	below ground surface
BV	bed volume
$CaCO_3$	calcium carbonate
Calgon	Calgon Carbon Corporation
CRS	Custom Resin Regeneration Service
CWA	Clean Water Act
DI	deionized
DO	dissolved oxygen
DPH	Department of Public Health
ESTCP	Environmental Security Technology Certification Program
$FeCl_3$	ferric chloride
$FeCl_4^-$	tetrachloroferrate
FWC	Fontana Water Company
gph	gallons per hour
gpm	gallons per minute
HCl	hydrochloric acid
IX	ion exchange
IIX	integrated ion exchange
MCL	maximum contaminant level
NaFC1	sodium chloride
NDEA	N-Nitrosodiethylamine
NDMA	N-Nitrosodimethylamine
NDPA	N-Nitrosodipropylamine
NAVFAC ESC	Naval Facilities Engineering Command / Engineering Service Center
NPC	net present cost
NSF	National Sanitary Foundation
O&M	operations and maintenance
ORNL	Oak Ridge National Laboratory
SDS	simulated distribution system
SDWA	Safe Drinking Water Act
SMCL	secondary maximum contaminant level
USEPA	U.S. Environmental Protection Agency
VOC	volatile organic compound

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1.0 EXECUTIVE SUMMARY

This report describes an evaluation of an integrated ion exchange (IIX) regeneration process for perchlorate treatment in drinking water. IIX combines three separate processes: conventional ion exchange (IX) with perchlorate selective resin for wellhead treatment of perchlorate contaminated water, regeneration of resin using tetrachloroferrate ($FeCl_4^-$) anion and then returning the resin to service, and the destruction or disposal of perchlorate recovered from the resin. The demonstration site was at an operating municipal water treatment plant owned by Fontana Water Company (FWC), in Fontana, CA. ARCADIS was the prime contractor (contract #W912HQ-06-C-004), with Calgon Carbon Corporation (Calgon) and Oak Ridge National Laboratory (ORNL) as partners. Funding and oversight were provided by the Environmental Security Technology Certification Program (ESTCP) and the Naval Facilities Engineering Command / Engineering Service Center (NAVFAC ESC). The project was funded under a special congressionally directed program to ESTCP for wellhead perchlorate treatment. This work was contracted through the Corps of Engineers in Alexandria, VA, and overseen by NAVFAC ESC. The program involved field activities at FWC and at a Calgon facility.

1.1 BACKGROUND

Perchlorate is a concern in drinking water because of its high solubility and mobility, known effects on thyroid hormone production, and treatment cost. The need for perchlorate treatment is especially acute in southern California's Inland Empire region. The Inland Empire's perchlorate plume is at least 6 miles long and impacts four cities' water supplies, resulting in impairment of 61,790 acre-feet per year (approximately 76.2 million cubic meters per year) of potable water.

IX is the only perchlorate treatment technology fully approved by the California Department of Public Health (DPH) for drinking water; biological reduction using a fluidized bed bioreactor has been conditionally approved. IX using a perchlorate-selective resin followed by resin disposal or destruction is the dominant market technology for perchlorate treatment. IIX involves the regeneration of perchlorate-selective resin using $FeCl_4^-$ anion and then returning the resin to service. Perchlorate in the spent $FeCl_4^-$ regeneration solution is subsequently destroyed or disposed of as a liquid waste stream. IIX will treat perchlorate in a more cost effective manner and may reduce the amount of purge water required during resin installation.

This IIX demonstration project included four perchlorate loading cycles to saturation using a 150 gallons per minute (gpm) wellhead treatment unit and a single batch of perchlorate-selective resin. After the perchlorate loading cycle, the resin was regenerated at an off-site facility using $FeCl_4^-$ solution. The modestly perchlorate-impacted fraction of the regenerant from each event was re-used in subsequent regeneration events without further treatment. A portion of the highly impacted fraction was used to evaluate chemical perchlorate reduction with ferrous chloride in a high pressure, high temperature pilot-scale reactor.

1.2 OBJECTIVE OF THE DEMONSTRATION

The general objective of the project is to demonstrate a reliable, more cost-effective method of treating low concentration perchlorate in drinking water supplies using IIX. Water treatment performance was demonstrated at 150 gpm (9.4×10^{-3} cubic meters per second [m^3/s]) for

approximately one year at an existing drinking water supply treatment facility. The principal goal was to demonstrate that regenerated resin could achieve California maximum contaminant level (MCL) for perchlorate:≤ 6 μg/L. An additional goal was for the regenerated resin effluent to maintain concentrations at or below MCL and secondary maximum contaminant level (SMCL) for nitrate and Title 22 metals, and at or below notification level, 10 nanograms per liter (ng/L), for nitrosamines. The performance of IIX was evaluated through several cycles to demonstrate the effectiveness and stability of the regenerated resin; the demonstration goal was to achieve 80-120% of virgin resin performance for treatment volume at breakthrough and perchlorate mass removal with regenerated resin.

1.3 REGULATORY DRIVERS

Throughout the United States, perchlorate standards or advisory levels are still evolving and currently range from 1 to 18 micrograms per liter (μg/L). No enforceable federal standard has yet been established. The U.S. Environmental Protection Agency (USEPA) has made a preliminary determination that setting a national drinking water standard for perchlorate is not justified under the terms of the Safe Drinking Water Act (SDWA). In January 2009 USEPA issued an interim health advisory level for perchlorate in drinking water of 15 μg/L. The California DPH finalized an MCL for perchlorate in drinking water of 6 μg/L on October 18, 2007. California DPH further requires that any treatment technology used in drinking water applications must have National Sanitary Foundation (NSF) 61 certification.

1.4 DEMONSTRATION RESULTS

IIX produced water comparable to that produced with the baseline technology—single-use perchlorate-selective resin treatment. No degradation in perchlorate-selective resin performance or impacts from metals carryover was found with IIX through three regeneration cycles despite re-using the $FeCl_4^-$ regenerant. Two effluent water samples contained measureable volatile organic compounds (VOCs) at concentrations below USEPA MCLs. The VOC source may include influent contamination, regeneration reagent contamination, or residuals from regeneration facility construction. IIX was not found to increase nitrosamines or other semivolatile organic compounds in treated water.

Perchlorate-loaded resin was regenerated in a multistep process utilizing $FeCl_4^-$ as the principal regenerant. The bulk of the perchlorate and nitrate were eluted within the first two bed volumes (BVs) of $FeCl_4^-$ regenerant. Pretreatment procedures were able to remove naturally occurring uranium from the resin prior to perchlorate regeneration. Post-regeneration procedures were able to return the resin to service with no significant water quality impact on initial wellhead effluent following a regeneration cycle.

Parametric destruction tests of spent $FeCl_4^-$ regenerant indicate pseudo first-order reduction of perchlorate using ferrous iron. Higher temperatures and residence times were associated with higher destruction efficiencies. Simulated perchlorate destruction tests routinely achieved greater than 95% destruction efficiency. High nitrate concentrations caused gas generation that led to process difficulties. VOCs were produced in destruction reactions.

1.5 STAKEHOLDER/END-USER ISSUES

IIX technology is currently licensed to Calgon from ORNL. Commercialization is expected to proceed using off-site regeneration at regional facilities contracted with lead times similar to new resin purchases. Operational differences between single-use resin and IIX will be isolated to the regeneration vendor and thus won't affect the utility. Reductive perchlorate destruction requires further development prior to commercialization; the vendor is likely to initially commercialize IIX with incineration of the spent regenerant until the perchlorate destruction process can be optimized. Cost savings with this technology have been predicted but will ultimately depend on the market price of the regeneration service and resin purchases, as well as the sales/use tax treatment of these items.

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2.0 TECHNOLOGY

2.1 TECHNOLOGY DESCRIPTION

IIX as demonstrated consists of three major components: (1) treatment of perchlorate from a drinking water source with a perchlorate-selective resin; (2) off-site regeneration of the resin with $FeCl_4^-$ solution; and (3) destruction (or disposal) of the perchlorate in the regeneration solution with ferrous iron. After the resin is regenerated, it is reused for treatment of perchlorate in drinking water. A highly perchlorate-loaded portion of the used $FeCl_4^-$ regenerant solution is processed through a perchlorate-destruction reactor rendering it suitable for reuse as a regenerant.

ORNL developed the first perchlorate-selective, bi-functional anion exchange resins and licensed the technology to Purolite and Thermax. Perchlorate-selective resins have two quaternary ammonium functional groups, one having long chain alkanes for higher selectivity and one having shorter chain alkanes for improved reaction kinetics. These resins are the dominant technology used in California for perchlorate treatment of drinking water. A diagram of the treatment system is shown in Figure 1.

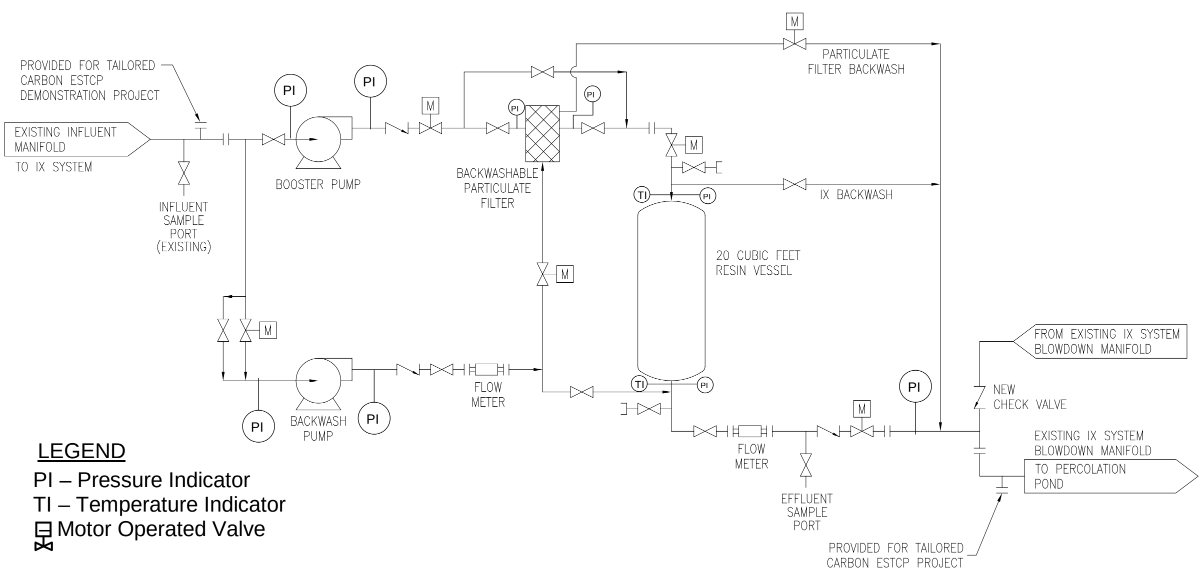

Figure 1. IX Process schematic used at FWC (backwash pump shown was not installed)**.**

Perchlorate-selective resins cannot be regenerated with conventional sodium chloride (NaCl) brine. ORNL developed a $FeCl_4^-$ regeneration technology (Gu et al., 2001; Gu et al., 2002a). $FeCl_4^-$ is a large, poorly hydrated anion similar to perchlorate. $FeCl_4^-$ anions, formed in a solution of ferric chloride ($FeCl_3$) and hydrochloric acid (HCl), can effectively displace perchlorate anion (ClO_4^-) from spent resins with as little as ~1 BV of regenerant. $FeCl_4^-$ anions subsequently dissociate in higher pH rinse water restoring the resin to its chloride (Cl^-) form. This technology

was previously demonstrated at the 20 ft^3 scale at Edwards Air Force Base (Gu et al., 2002a; Gu et al., 2003b). Calgon modified the process by adding a rinse step prior to $FeCl_3^-$ HCl regeneration to reduce the generation of impurities. A diagram of the regeneration and destruction technology processes is shown in Figure 2.

Perchlorate in the regenerant solution is chemically reduced to chloride with ferrous iron at a temperature of approximately 200 °C (392 °F) (Gu et al., 2002b; Gu et al., 2003a). Perchlorate is reduced and ferrous iron is oxidized, which replenishes or "regenerates" the $FeCl_4^-$ regenerant. The overall chemical reaction can be written as:

$$ClO_4^- + 8Fe^{2+} + 8H^+ \rightarrow Cl^- + 8Fe^{3+} + 4H_2O$$

The reaction proceeds at a high pressure, primarily to keep the water in a liquid phase. This process has been tested in a small batch system and both laboratory- and field-scale flow reactors at ORNL (0.1 mL/min up to ~1.5 gallons per hour [gph]) (Gu et al., 2003a).

2.2 ADVANTAGES AND LIMITATIONS OF THE TECHNOLOGY

Drinking water treatment with perchlorate-selective resin is currently the dominant perchlorate treatment technology. Although perchlorate-selective resins are more expensive than conventional IX resins, the service life is much greater than conventional strong base anion resins and greater than nitrate-selective resins. IIX will permit re-use of resin, saving resources to produce resin and potentially reducing operating costs. Re-use of resin also reduces nitrosamine and nitrosamine precursor residuals associated with installation of virgin resin. Regenerated resin has not yet been NSF-certified and thus cannot currently be used in drinking water applications.

Regeneration of the resin with $FeCl_4^-$ solution results in perchlorate concentrated in a small volume of regenerant, nominally 1 BV (<0.001% of treated water volume at FWC), compared to brine production at 1 to 5% of treated water volume for other resin regeneration applications (nitrate treatment, water softening, etc.). $FeCl_4^-$ regeneration will remove most other contaminants from the resin resulting in a buildup of metals in recycled $FeCl_4^-$ regenerant; contaminant buildup is managed by purging a portion of the $FeCl_4^-$ regenerant. The $FeCl_4^-$ regenerant requires special materials and handling since it is corrosive and produces corrosive vapors. However, the regenerant is handled only at the vendors' facility, not at the drinking water treatment facility.

Destruction of perchlorate with ferrous chloride solution restores the regenerant for reuse. The amount of ferrous chloride solution required determines the minimum purge required to maintain a constant inventory of regenerant. Nitrate in the regenerant increases reagent demand; the overall reaction with nitrate can be summarized as:

$$2NO_3^- + 10Fe^{2+} + 12H^+ \rightarrow N_2 + 10Fe^{3+} + 6H_2O$$

Presence of nitrate in the regenerant increases the ferrous chloride requirement and generates nitrogen gas. Alternative destruction technologies could be investigated including reduction with ethanol or propanol, incineration, or other appropriate disposal techniques. The destruction reactor must be able to resist corrosion in a high temperature, high pressure environment and the generation of gaseous by-products.

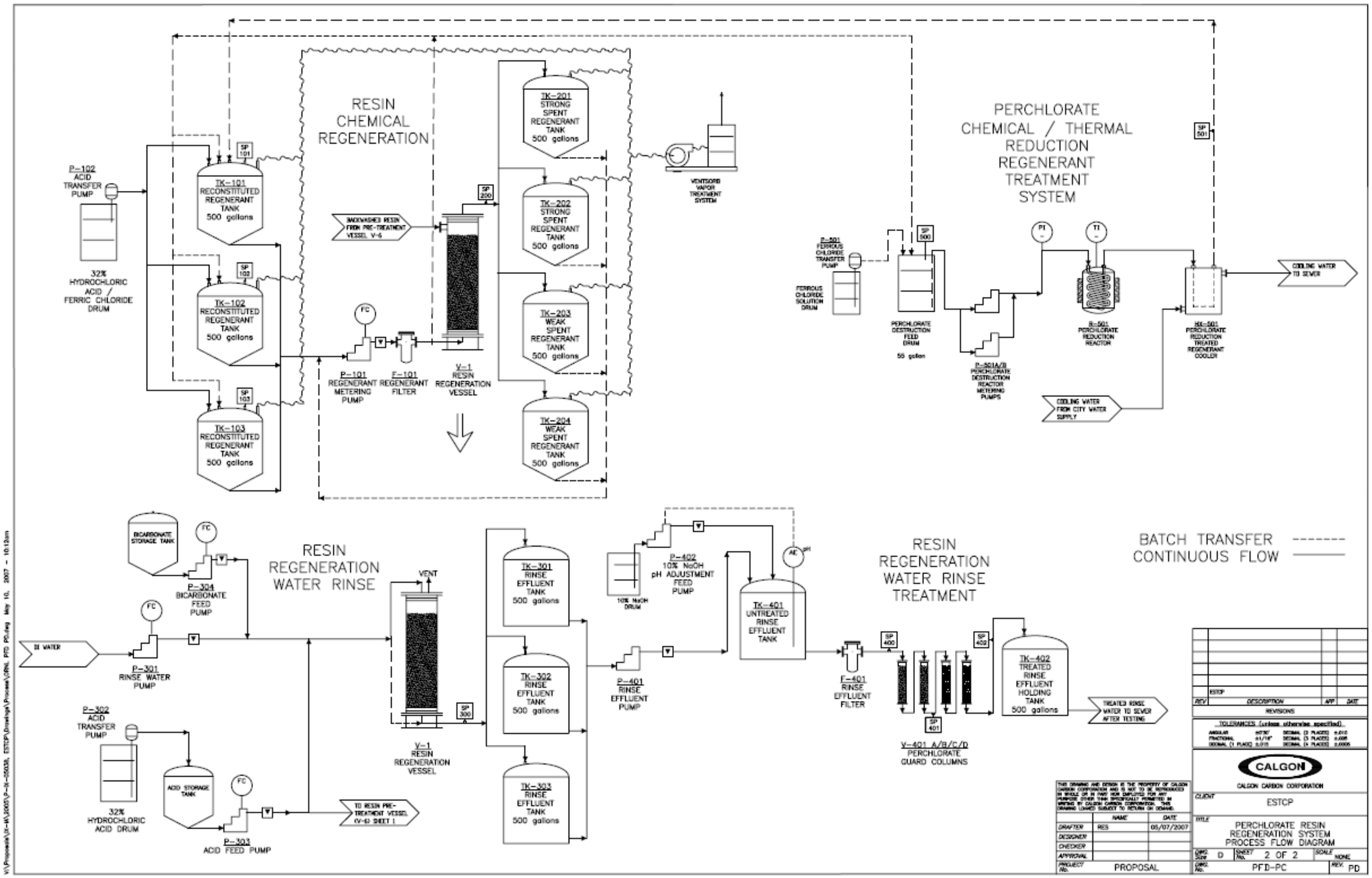

Figure 2. Diagram of regeneration, rinse, and destruction systems.

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3.0 PERFORMANCE OBJECTIVES

The overarching goal of the project was to demonstrate an IIX, regeneration, and destruction process for treating perchlorate in drinking water. Specific objectives and how they were met are summarized in Table 1.

Table 1. Performance objectives.

Performance Objective	Data Requirements	Success Criteria	Result
Quantitative Performance Objectives			
Meet perchlorate MCL	Perchlorate concentration in treated effluent	<6 µg/L perchlorate after 1 or more regeneration cycles	<2 µg/L at breakthrough; MCL not exceeded for >145,000 BV
Regenerated resin comparable to virgin resin	Perchlorate breakthrough	80-120% of 1st cycle	85-119%
	Perchlorate loading	80-120% of 1st cycle	83-90%
Regenerated resin produces acceptable nitrate, metals, and nitrosamine concentration	Treated effluent concentrations	Nitrate (as NO_3) ≤ 45 mg/L Metals ≤MCL, nitrosamines ≤10 ng/L;	No exceedances
Perchlorate destruction	Reactor influent and effluent perchlorate concentration	≥95% destruction	>99%
Limit uranium carryover	Uranium concentration in treated effluent	≤30 µg/L uranium after 1st cycle	No exceedances
Qualitative Performance Objectives			
Reduced treatment costs	Life-cycle cost	≥25% reduction	12% reduction
Identify and assess scaling parameters	Operations assessment	Identify scale-up issues	Flow and pressure issues identified
Identify, assess and overcome integration issues	Treated effluent concentrations	Produce acceptable drinking water	Potential VOC carryover from regeneration
Time to saturation of regenerated resin	Volume at perchlorate saturation	Document resin degradation	No degradation observed
Rinse volume requirement (during regeneration)	Volume required	Determine # of rinse bed volumes needed	Rinse volumes measured, reductions in rinse volume possible
Document required neutralization or other rinse water treatment requirements	Mass and volume of neutralization agent used	Determine reagent requirements for discharge	Alternate disposal used; costs estimated.
Regeneration process efficiency	Perchlorate elution profile	Verify <6 BVs regenerant require treatment	~2 BVs removed per cycle with no degradation
Determine necessary regenerant purge rate	Determine accumulation of anions, hardness, organic matter, uranium, particulate matter	Identify concentrations impairing regeneration	No adverse effects encountered
Determine optimum perchlorate destruction conditions	Perchlorate destruction under parametric testing	Determine flow rate and temperature optima	No optimum in flow rate or temperature
Ease of operations and maintenance (O&M)	Field technician feedback	Same or better for operator versus conventional system	O&M same except less purging is necessary
Regenerant readjustment requirement	Amount of reagent required	Document the amount of readjustment required to the recycled regenerant to meet acceptance criteria.	Some information obtained

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4.0 SITE DESCRIPTION

4.1 SITE LOCATION AND HISTORY

The field demonstration was performed at the existing FWC perchlorate treatment facility located in Fontana, CA, adjacent to groundwater production wells FWC-17B and FWC-17C. The FWC full-scale perchlorate treatment system at the same site utilizes one-pass (non-regenerable) IX that consists of five parallel trains of lead-lag vessel pairs (a total of 10 IX vessels), and has a maximum treatment capacity of approximately 5000 gpm (0.3 m^3/s). Production wells FWC-17B and FWC-17C pump water through the treatment system and then to FWC's distribution system reservoir. Currently, the IX system utilizes Purolite A-530E resin, the same resin that was used during the demonstration. The site also has a National Pollution Discharge Elimination System-permitted percolation pond that is used to discharge water generated during resin change-outs and well blowdown. A site location map is provided as Figure 3.

4.2 SITE GEOLOGY/HYDROGEOLOGY

The demonstration site houses two groundwater production wells, a drinking water reservoir, a percolation pond, and a perchlorate treatment system. Groundwater is extracted from the Chino formation. Based on U.S. Geological Survey, the site is likely to be high in dissolved oxygen (DO) (>50% of saturation), with dissolved organic carbon below 1 mg/L, phosphorous below 0.04 mg/L and alkalinities between 130-180 mg/L as calcium carbonate ($CaCO_3$). Production wells are screened from 500 ft to 860 ft below ground surface (bgs) and from 500 ft to 920 ft bgs. Wells are managed for production and nitrate concentration using pumping time and inflatable packers.

4.3 CONTAMINANT DISTRIBUTION

The site sits above a large regional perchlorate plume that is unrelated to past or current site operations. It is located in a light industrial/residential area of Fontana, CA. Perchlorate concentrations have ranged from 8.2 to 24 µg/L for well 17B and 4.0 to 19 for well 17C from June 1998 to January 2007.

Figure 3. Site map, FWC-17B and FWC-17C.

5.0 TEST DESIGN

5.1 CONCEPTUAL EXPERIMENTAL DESIGN

The overall objective of the project was to demonstrate perchlorate treatment of drinking water using the IIX process. The demonstration utilized Purolite A-530E bi-functional perchlorate-selective resin to treat perchlorate at FWC. The resin was saturated with perchlorate and removed from the site for regeneration at Calgon's facility in Pittsburgh, PA. The resin was regenerated with $FeCl_4^-$ solution and returned to service at FWC. This process was repeated for a total of four cycles of perchlorate loading. The $FeCl_4^-$ solution was re-used in each regeneration cycle. A highly contaminated portion of the $FeCl_4^-$ solution was treated in a destruction reactor to chemically reduce the recovered perchlorate. A portion of the treated $FeCl_4^-$ solution was returned to the $FeCl_4^-$ solution inventory prior to commencing the third regeneration cycle.

Water quality parameters were monitored at the influent and effluent of the demonstration unit during perchlorate treatment. The perchlorate removal performance of newly manufactured resin was compared to the performance with regenerated resin. Operating the single resin bed to perchlorate saturation (defined as an effluent perchlorate concentration of 90% or more of the influent perchlorate concentration) provides loading information comparable to commercial dual-bed systems. Re-use of the same resin permits the evaluation of IX resin degradation from the regeneration process. Other water impacts were also monitored to address concerns with contaminant carryover from the regeneration cycle.

A simplified flow chart of the regeneration and destruction operations is shown in Figure 4. Eluent chemistry during the regeneration cycle was monitored to determine the elution profile of perchlorate and evaluate regenerant volume savings potential within the regeneration process.

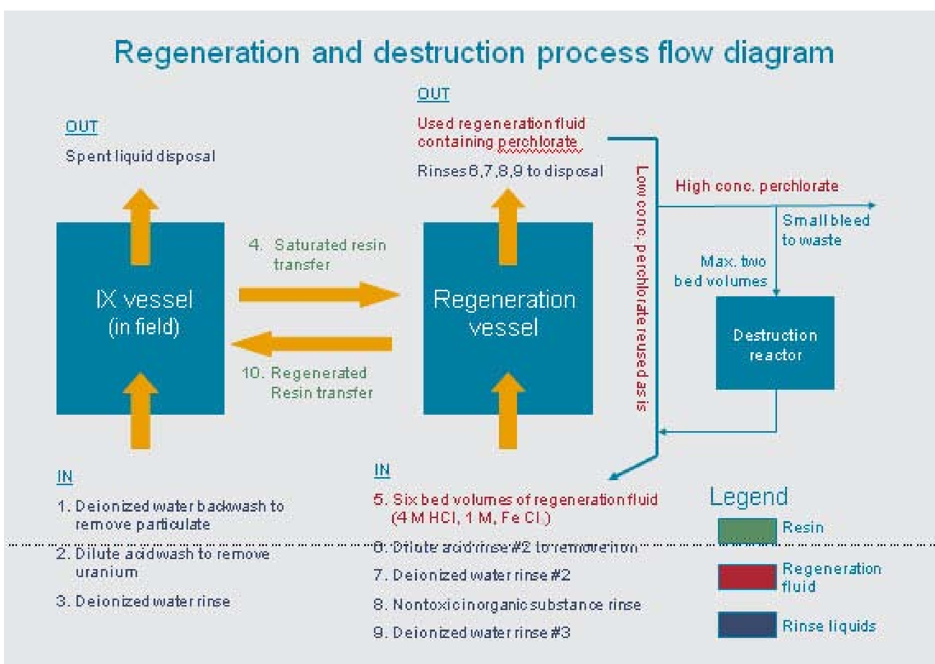

Figure 4. Simplified process flow diagram of regeneration and destruction.

$FeCl_4^-$ with high perchlorate concentration was selected for perchlorate destruction testing. Parametric destruction tests were performed monitoring water quality parameters varying temperature and flow rate at constant stoichiometry. Optimum reaction conditions were used in longer tests to demonstrate the commercial destruction feasibility.

5.2 BASELINE CHARACTERIZATION

Baseline was defined as the untreated water from wells FWC-17B and/or FWC-17C, the influent to the treatment system. Influent water quality was routinely monitored along with the effluent during pilot system treatment. Well pumping rates were controlled by the needs of the FWC and its customers. Variability in the concentrations of nitrate and perchlorate in these wells is likely a function of regional hydrogeology, the pumping rates, and well packer depth adjustments made to these supply wells. The chemistry was typical of groundwater that is treated in the California Inland Empire; however, the average perchlorate concentration is slightly lower, and the average nitrate concentration is slightly higher than at other perchlorate treatment systems in the area. Influent perchlorate concentrations were monitored to determine resin loading. In addition, influent concentrations of perchlorate, nitrate, and uranium were of particular interest with respect to identifying any rollover effects. Influent nitrosamines were monitored to help identify potential leaching issues with regenerated resins. A summary of these influent parameters is provided in Table 2. Additional influent water chemistry data is presented in the final report for this project. Influent perchlorate concentration, shown in Figure 5, appears to be time dependent over the time intervals associated with each IX perchlorate loading cycle. Perchlorate variability impacts performance parameters including perchlorate loading and saturation volume. Influent nitrate was managed by FWC well activity.

Table 2. Critical influent water characteristics.

	Minimum	Maximum	Median
Perchlorate	<2.0 µg/L	11.0 µg/L	7.7 µg/L
Nitrate	29 mg/L as NO_3	40 mg/L as NO_3	37 mg/L as NO_3
Uranium	1.6 µg/L	2.6 µg/L	2.2 mg/L
NDMA	<2.0 ng/L	32 ng/L	<2.0 ng/L
NDEA	<2.0 ng/L	<2.0 ng/L	<2.0 ng/L
NDPA	<2.0 ng/L	<2.0 ng/L	<2.0 ng/L

NDMA – N-Nitrosodimethylamine
NDEA – N-Nitrosodiethylamine
NDPA – N-Nitrosodipropylamine

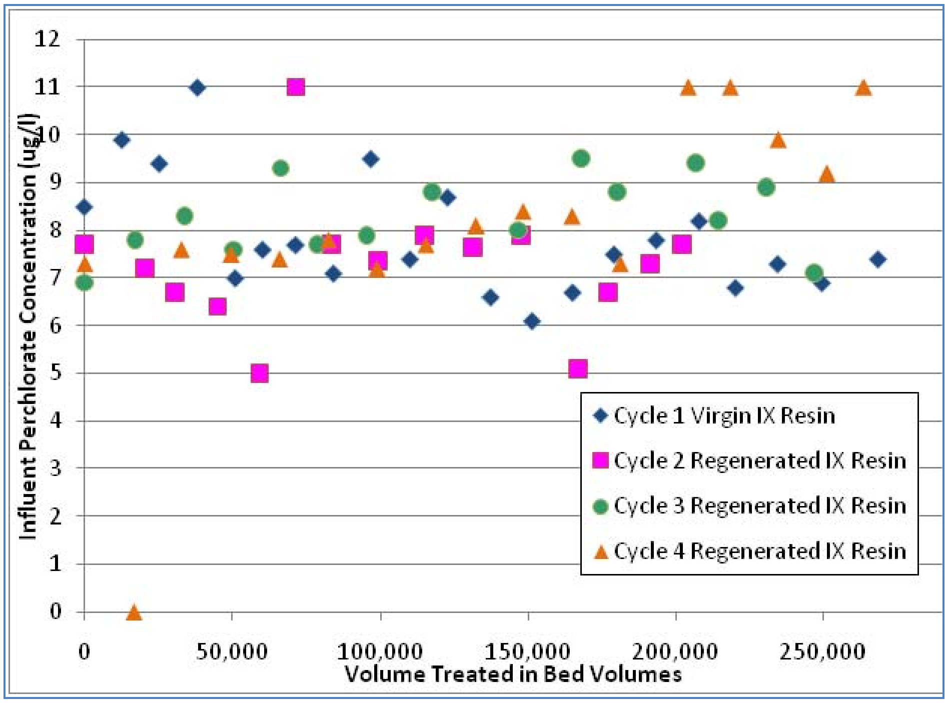

Figure 5. Influent perchlorate concentrations at FWC wellhead IX treatment unit.

5.3 TREATABILITY OR LABORATORY STUDY RESULTS

The primary technologies being tested did not undergo any treatability or laboratory studies specific to this demonstration site. However, ARCADIS conducted simulated distribution system (SDS) testing on the IX system effluent on two occasions to determine the potential for disinfection by-product generation. Samples were dosed with excess bleach as a disinfectant and incubated at 21 °C for 1 day or 7 days. Treatments were then analyzed for Total Organic Halides (SM5320B), haloacetic acids (USEPA 552.2), trihalomethane (USEPA 524.2), and nitrosamines (USEPA 521). During the second SDS test, total organic halides and trihalomethane, principally chloroform, were slightly elevated compared to the control treatment. No nitrosamines were detected.

5.4 FIELD TESTING

A concrete pad was installed at the FWC site to contain the demonstration system. The treatment system was installed at FWC, loaded with resin, and backwashed to remove particulate and residual nitrosamines. The off-site regeneration system and perchlorate destruction module were fabricated and operated in Calgon's facility in Pittsburgh, and did not require any on-site setup for this demonstration

A Gantt chart detailing system operation window is shown in Figure 6.

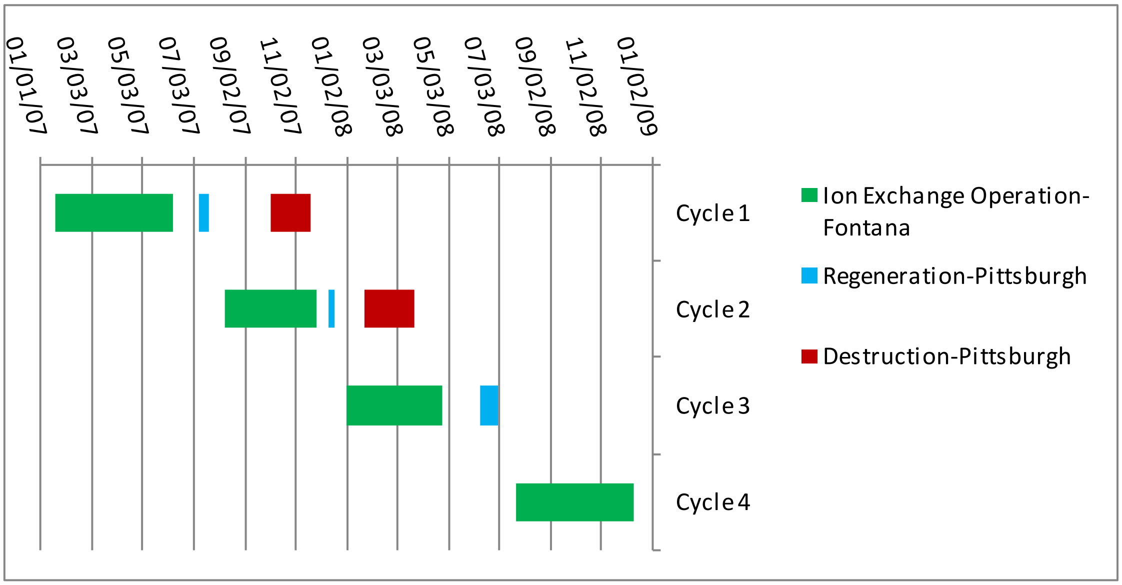

Figure 6. Project operational Gantt chart.

The demonstration treatment system operated between January 17, 2007, and December 11, 2008—approximately 474 operational days, treating over 144 million gallons of perchlorate-impacted groundwater. Groundwater was treated by the demonstration system until the resin was saturated with perchlorate, at which time the IX vessel (and resin) was shipped to Calgon's facility in Pittsburgh for resin regeneration and perchlorate destruction. Following resin regeneration, the IX resin was reloaded into the IX vessel, returned to FWC, and resumed operation for another load/regenerate cycle. This cycle was repeated three times during the demonstration period. During the demonstration, the system operated at 95% uptime efficiency. The majority of the demonstration system's downtime was not directly attributable to the demonstration system but was mainly caused by shutdowns of the larger FWC system that hosted the demonstration. The primary causes of downtime attributable to the IIX demonstration system was caused by the IX vessel piping.

5.5 SAMPLING METHODS

Performance of IIX was evaluated in three separate phases of operation: treatment, regeneration, and perchlorate destruction. Samples were subjected to a variety of chemical analyses including perchlorate, metals, nitrosamines, VOCs, and other general water quality parameters. During treatment, water was sampled from the influent and effluent of the demonstration system. In addition, certain engineering parameters were recorded during treatment including flow rate and total treated water volume.

Water was sampled during regeneration at the bed influent, effluent, and from effluent receiving containers for composite samples. Again, flow rate and total volume or mass were critical engineering parameters that were recorded.

Composite water samples were collected from the destruction reactor feedstock, from reactor effluent receiving containers, and directly from the reactor effluent for perchlorate destruction tests.

5.6 SAMPLING RESULTS

5.6.1 IX Unit Process Sampling Results

The effluent perchlorate concentrations from the IX treatment unit are shown in Figure 7 through all four resin loading cycles. The effluent concentrations remained below the reporting limit for a significant treatment volume after installation of virgin IX resin and after each installation of regenerated IX resin (typically>100,000 BVs); non-detects have been plotted as 0.0 µg/L perchlorate. Perchlorate concentration of the effluent gradually increases with increased treatment volume. The breakthrough curves for the regenerated resins are essentially indistinguishable from that of the virgin resin and from each other.

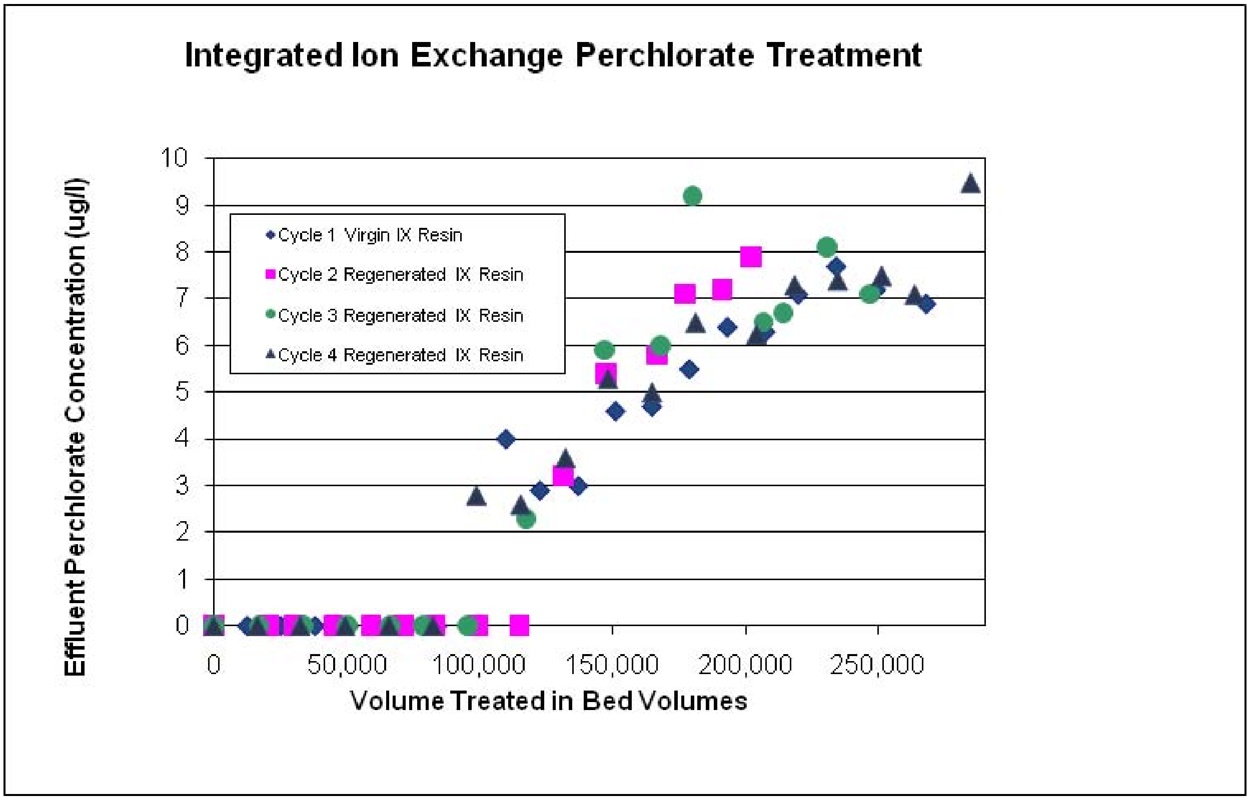

Figure 7. Effluent perchlorate concentration at FWC IX treatment unit.

No exceedances of nitrate, Title 22 metals, or nitrosamine MCL or notification levels were observed in water treated with regenerated resin. Uranium exhibited breakthrough similar to perchlorate; no evidence of chromatographic rollover was observed for uranium or other metals. VOCs were found in the treated water at the start of the second treatment cycle; this contamination is likely an artifact from fabrication of the regeneration unit since it occurred after the first regeneration cycle.

5.6.2 Regeneration Unit Process Sampling Results

The resin from the treatment process was shipped in the treatment vessel to Calgon's Pittsburgh facility for regeneration. Regeneration is a multistep process including a deionized (DI) backwash, a dilute acid backwash, resin transfer to the regeneration vessel, regeneration with

$FeCl_4^-$, a dilute acid rinse, a DI water rinse, a nontoxic inorganic substance rinse, a final DI rinse, and resin transfer back to the treatment vessel.

Uranium was largely removed from the resin during the dilute acid backwash and co-eluted with sulfate. This step limited the buildup of uranium in the $FeCl_4^-$ regeneration solution. Most of the uranium was recovered in the first 4 BVs of dilute acid.

Perchlorate elution profiles during $FeCl_4^-$ regeneration are shown in Figure 8. In all three cycles, there is a lag in perchlorate elution followed by a sharp increase in perchlorate concentration. The rengerant volume at which this peak occurs is reasonably consistent at ~100 gallons or 1 BV. Nitrate co-eluted with perchlorate with a similar profile. Iron in regenerant solution lags similar to perchlorate followed by breakthrough and saturation.

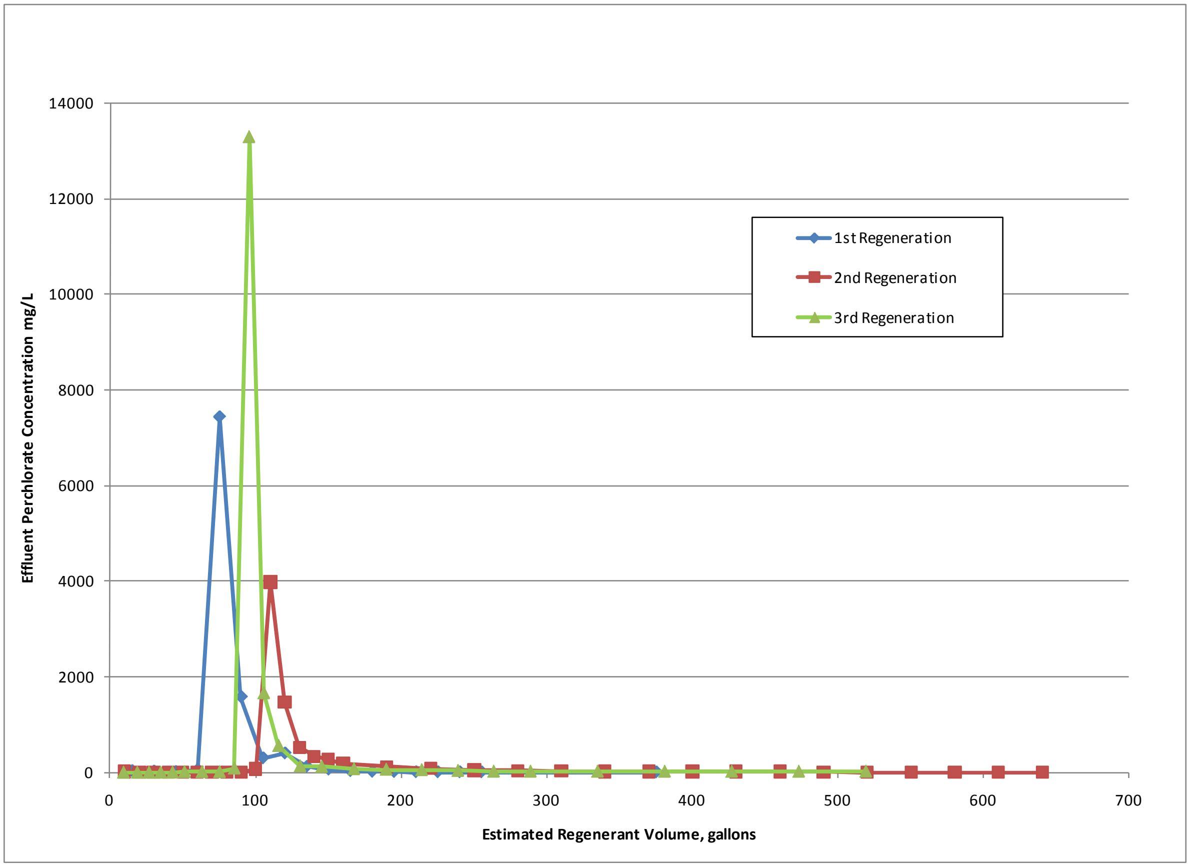

Figure 8. Perchlorate elution during tetrachloroferrate regeneration.

After $FeCl_4^-$ regeneration, the resin was rinsed with 600 gallons of dilute acid. Total iron concentration decreased with rinse volume but was still significant at the end of the rinse (sometimes >1 g/L). Either a slower dilute acid rinse or more rinse volume is expected to improve iron washout.

Nitrosamines were found in various regeneration samples. The demonstration was not designed to determine whether these nitrosamines were carried over from groundwater treatment or generated in the regeneration process. Regeneration did not, however, impact nitrosamines in treated water at FWC.

5.6.3 Destruction Unit Process Sampling Results

A series of parametric destruction tests was performed with the $FeCl_4^-$ regenerant from both the first and second regeneration cycle. As shown in Figure 9, perchlorate concentration in the reactor effluent decreased with increasing reaction temperature and with increasing residence time. Residence time increases as flow rate decreases in the fixed volume reactor. Nitrate concentration was reduced through the destruction reactor, along with total organic carbon and nitrosamines, but in a less consistent manner. Gas production created operational difficulties controlling flow rate and residence time for some tests. Some VOCs including chloromethane, 1,2-dichloroethane, and bromomethane appear to be created in the destruction reactor.

A series of perchlorate destruction runs were performed at ~190 °C with $FeCl_4^-$ regenerant from the second regeneration cycle. Feedstock was managed to limit nitrate concentration and control flow disturbances caused by gas production. Flow rate remained difficult to control causing extreme variation in flow rate during some perchlorate destruction runs. Typical perchlorate destruction exceeded 99%.

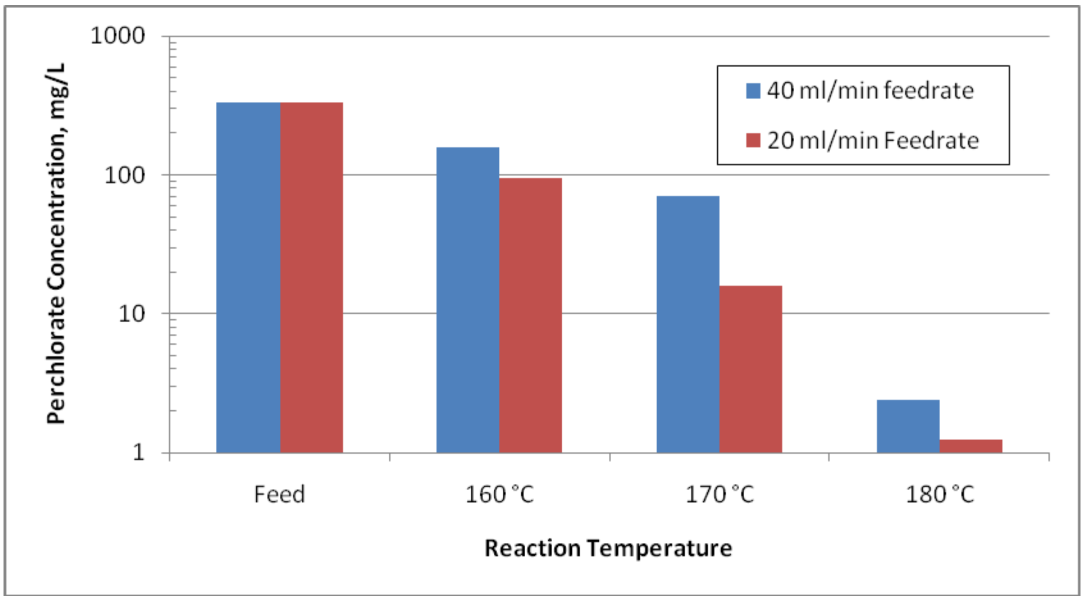

Figure 9. Parametric destruction reactor test results from second regeneration cycle.

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6.0 PERFORMANCE ASSESSMENT

Many performance parameters are evaluated in terms of the volume of water treated or the volume fed during regeneration. Volumes were routinely normalized by the volume of resin in the treatment or regeneration vessel. The volume of resin changed slightly due to losses, generally attributable to loading events not inherent to the IIX process. The residence time in the destruction reactor was determined by the ratio of the fixed volume of the reactor to the reactor effluent flow rate.

6.1 TREATMENT PERFORMANCE

The overall objective for the perchlorate treatment phase was to produce acceptable water quality with IIX. This objective was evaluated comparing all perchlorate treatment effluent analytical results to MCLs or notification levels. Since the treatment unit was intentionally operated to perchlorate saturation using a single resin bed, this objective for perchlorate was evaluated until perchlorate breakthrough occurred. For perchlorate, the treatment IX vessel maintained effluent below analytical reporting limits (2.0 µg/L) for more than 82,000 BVs and below the MCL (6 µg/L) for more than 146,000 BVs. No MCL exceedances were observed for nitrate, Title 22 metals, or nitrosamines during treatment with regenerated resin through perchlorate saturation; nitrosamine washout was observed (which is common with some types of virgin resins). In addition, the virgin resin had one sample with aluminum and one with selenium exceedances.

The performance of the regenerated resin was further compared to the performance of virgin resin with respect to perchlorate breakthrough, saturation, and perchlorate loading at saturation. Since the effluent perchlorate breakthrough concentration at FWC, which is defined as 10% of influent perchlorate concentration, was less than the analytical reporting limit, breakthrough volume was estimated based on the effluent sampling event immediately preceding the first sample with a quantifiable perchlorate concentration. The precision of this estimate is controlled by the demonstration sampling schedule. The perchlorate breakthrough volume with regenerated resin was within the demonstration objective of ±20% of the virgin resin breakthrough for all treatment cycles. Perchlorate saturation was similarly defined by an effluent perchlorate concentration ≥90% of the influent perchlorate concentration. Perchlorate saturation with regenerated resin occurred after treating between 167,000 and 264,000 BVs, compared to virgin resin after 208,000 BVs. No degradation in perchlorate saturation performance of the regenerated resin was discernable in this demonstration.

Perchlorate loading, the mass of perchlorate removed during treatment, was determined by integrating the difference between the influent and effluent concentration versus volume curves. Integration was performed using a trapezoidal approximation; the difference between coincident influent and effluent sample concentrations was averaged for successive perchlorate sampling events and multiplied by the volume treated between these sampling events. For this mass removal calculation, a concentration of 0.0 µg/L was used for all perchlorate concentrations falling below the method's reporting limit of 2.0 µg/L. Resin mass loading through each treatment cycle is summarized in Table 3. Virgin resin removed 515 g of perchlorate before saturation. Regenerated resin achieved loading between 83 and 90% of the virgin resin loading before saturation. This analysis demonstrates that regenerated resin meets the performance

objective of 80 to 120% of virgin resin perchlorate loading before saturation through three resin regeneration cycles.

Table 3. Perchlorate loading at FWC IX treatment unit.

	Cycle 1	Cycle 2	Cycle 3	Cycle 4
Loading before breakthrough, g	364	359	308	201
Loading before saturation, g	515	425	448	465
Final loading, g	516	429	486	483
Normalized final loading, g/L	1.19	1.01	1.19	1.18

6.2 REGENERATION PROCESS PERFORMANCE

Regeneration operations were primarily focused on producing a resin that would ensure good treatment performance. A number of qualitative performance objectives were evaluated to streamline the regeneration process and minimize waste.

A major regeneration objective was to verify that only a small amount of $FeCl_4^-$ regenerant requires perchlorate destruction or disposal as a liquid waste stream. Six BVs of $FeCl_4^-$ regenerant was used to recover perchlorate, and this was clearly sufficient for regeneration. The perchlorate eluted in the $FeCl_4^-$ regeneration step was calculated by multiplying analytical results from composite samples collected from the receiving container by the volume in the receiving container, and then adding the mass contained in each container. The perchlorate elution during the $FeCl_4^-$ regeneration steps are illustrated in Figure 10. The first 50 to 100 gallons of eluent during the $FeCl_4^-$ regeneration step contained very little perchlorate, iron, or acidity. The perchlorate then elutes very rapidly over the next 50 to 100 gallons. It is quite possible to reduce the $FeCl_4^-$ feed volumes in future scale-ups or production service. The vast majority of the perchlorate recovered from regenerating the IX resin elutes in the first 2 BVs of $FeCl_4^-$ regenerant solution. During this demonstration, ~2 BVs of $FeCl_4^-$ regenerant was removed from service each cycle for perchlorate destruction and/or disposal; the remaining $FeCl_4^-$ regenerant remained in service for the next regeneration cycle. The success of the perchlorate treatment demonstrates that the residual perchlorate in the $FeCl_4^-$ regenerant resulting from re-using the regenerant did not adversely impact performance.

Despite the re-use of $FeCl_4^-$ that was processed through the perchlorate destruction reactor, some volume of $FeCl_4^-$ purge is required to maintain a constant $FeCl_4^-$ inventory from ferrous iron addition or to maintain contaminants at an acceptable level. The determination of an optimum purge volume in this demonstration was complicated by variable inventory over the duration of the demonstration, and iron carryover to the post-regeneration acid rinse. Furthermore, the limited number of cycles did not produce contaminants at a level that interfered with treatment, regeneration, or destruction processes.

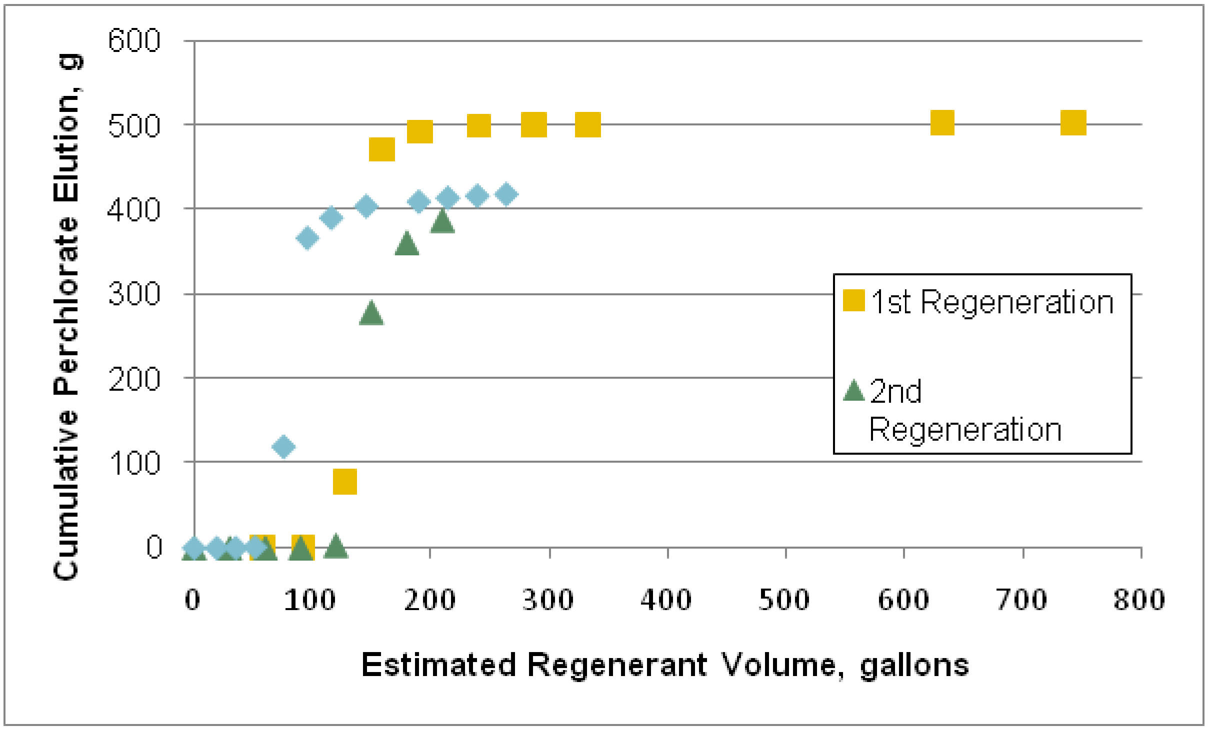

Figure 10. Perchlorate mass elution during regeneration.

Refinements to the regeneration process were made during the demonstration as a result of the elution profile of various reagents and from operational experience. Additional optimization can be expected based on additional data and experience. One rinse step in the process was removed entirely. The dilute acid wash, designed to strip uranium from the resin, may be reconsidered depending on the ultimate perchlorate disposal procedure that is commercialized. Further, the data suggest that additional or slower acid rinse after $FeCl_4^-$ regeneration could reduce subsequent rinse requirements; this acid rinse may be recyclable within the regeneration process.

6.3 PERCHLORATE DESTRUCTION

The perchlorate destruction process was principally evaluated with perchlorate destruction efficiency, the percentage of perchlorate destroyed. Perchlorate destruction efficiency was calculated as:

$$\text{Destruction Efficiency} = \left(1 - \frac{\text{Effluent Perchlorate Concentration}}{\text{Influent Perchlorate Concentration}}\right) \bullet 100$$

Parametric destruction tests demonstrated that >95% destruction efficiency could be achieved at 180 °C reaction temperatures with a 1.5 hour residence time. Also >99% destruction efficiency could be achieved at 190 °C reaction temperatures with a 1.1 hour residence time. Semicontinuous destruction runs at ~190 °C, performed to approximate commercial operation, achieved 73.6% to >99.7% destruction efficiency, with a median efficiency of 99.2% under a range of residence times, perchlorate concentrations, and nitrate concentrations. It is clear that the objective of 95% perchlorate destruction can be maintained over a wide range of feed

compositions, although some management of nitrate concentration and ferrous iron stoichiometry will be required.

Parametric destruction tests were performed to identify optimum reaction conditions. No physical optimum was identified, and identifying an economic optimum was beyond the scope of this demonstration. As a tool for evaluating economic optimum, a pseudo first-order reaction rate constant was determined for the parametric destruction runs with less than 95% destruction efficiency in order to compare with previously published destruction kinetics.

$$k = \frac{\ln[ClO_4^-(inlet)/ClO_4^-(outlet)]}{\text{Residence Time}}$$

As shown in Figure 11, the rate constants from the demonstration-scale tests agree well with the published rate constants from smaller-scale work (Gu et al., 2003a).

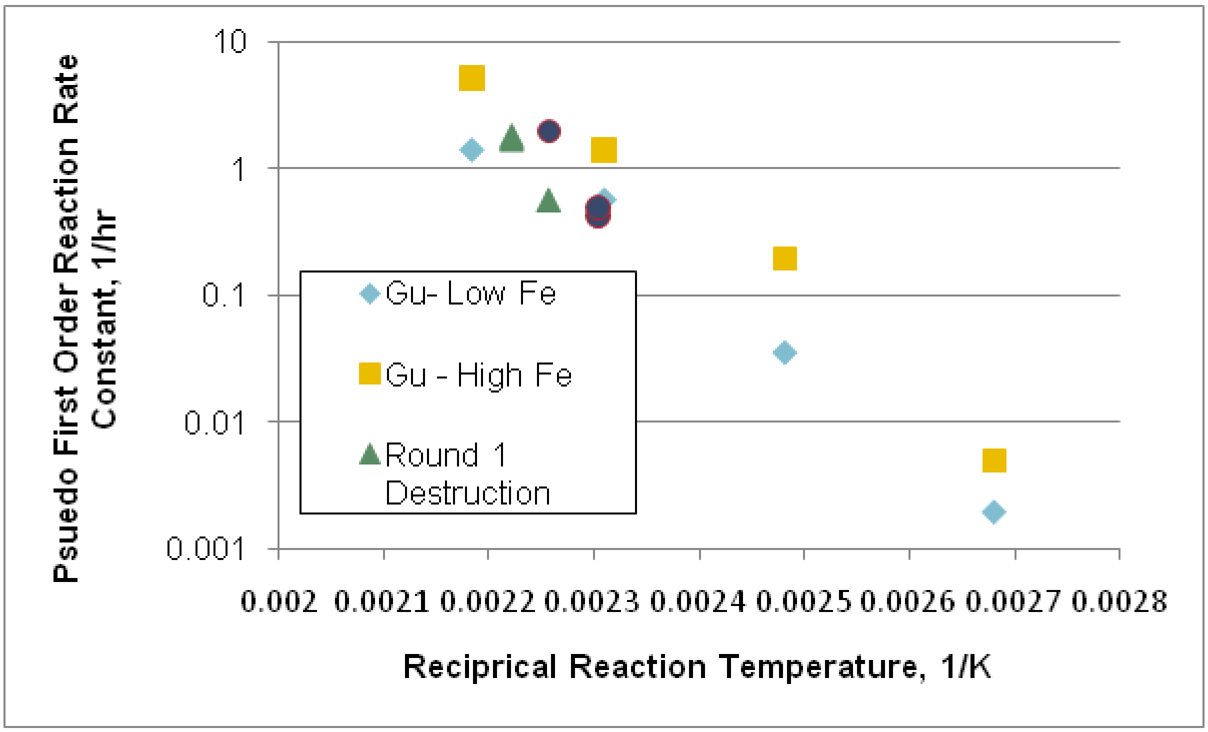

Figure 11. Perchlorate destruction reaction rate constant as a function of temperature.

6.4 IMPLEMENTATION

From the perspective of a water treatment plant, the O&M characteristics of an IIX system are expected to be the same as those of a single-use IX system. The regeneration and destruction technologies are compartmentalized with the commercialization vendor. The technologies are expected to be interchangeable for the water plant, permitting a change in technology as market conditions dictate. The only operational difference identified is the reduction of nitrosamines

(NDMA, etc.) contamination with IIX and thus the ability to avoid extensive purging prior to placing regenerated resin back in service.

A cost model for this technology was produced to compare it with single-use IX, and is presented in Section 7 for a 30-year operational period. For the particular application modeled, the total net present cost (NPC) for the IIX technology was $16.4 million, and the total NPC for the single-use IX was $18.6 million. NPCs were calculated using a real interest rate of 2.7%, supplied by Office of Management and Budget. Thus the IIX technology appears to be approximately 12% less expensive. Thus a cost savings was demonstrated, but not as large as the 25% goal stated in the demonstration plan.

The perchlorate-selective resin component of the technology was already operational at full-scale before this demonstration; scale-up is generally as simple as placing multiple vessel pairs in parallel. A comparison of the bench- and pilot-scale work for resin regeneration shows very good broad agreement in performance characteristics—percent recovery of perchlorate, number of cycles of resin reuse, and BVs required for perchlorate elution. This suggests that the regeneration technology is readily scalable. Although reaction kinetics from this demonstration largely agree with previous work, substantial operational difficulties in flow control, etc. were experienced with the perchlorate-destruction reactor. The authors believe that the destruction reactor would require additional substantial refinement for it to operate routinely in commercial service at the demonstration or larger scale. An alternate perchlorate destruction technology, such as incineration of the regenerant, can be used pending commercialization of the destruction reactor without major impacts to the IIX technology. The lack of major impact is attributable to the small quantity of regenerant purge that is required by the destruction reactor and the sharp elution profile of the $FeCl_4^-$ regenerant.

All three unit processes were integrated in this demonstration. Some issues were identified that could be attributed to the integration of the technologies:

- Gas generation was observed during perchlorate destruction unit operation, likely attributable to nitrate accumulation in the regenerant solution from high nitrate in the FWC groundwater.
- Nitrosamines were found in significant concentrations in the $FeCl_4^-$ regenerant solution and in rinses following the $FeCl_4^-$ regeneration. Although nitrosamines were found in the regenerant solutions, there was no evidence that nitrosamines carried through with the resin to be found in the treatment system effluent stream.
- Halogenated VOCs appear to have been formed in the perchlorate destruction reactor. Concentrations in IX effluent were below USEPA MCLs.

- In addition to the VOCs generated in the perchlorate destruction reactor, another source of VOCs is present since VOCs were found in $FeCl_4^-$ solutions that had not passed through the destruction reactor. Furthermore, chloromethane was also present in the first cycle $FeCl_4^-$ effluent from the regeneration vessel. It is unclear whether the VOCs are:
  - Present below detection limits in the groundwater and concentrated on the resin during field treatment and then removed during regeneration
  - Entered the process as contaminants in the reagents or equipment used
  - Were created chemically during the ion-exchange or regeneration process.

VOCs were only an issue in the field after the first regeneration, suggesting this problem could be a one-time occurrence related to the construction of the regeneration facility.

7.0 COST ASSESSMENT

7.1 COST MODEL

The cost model for IIX used in drinking water applications is built upon the design practices and experience of single-use IX installations. IIX technology is an alternative to resin replacement in a conventional single-use IX application. There is no difference in the design and operation of the field portion of the technology with either single-use resin or IIX. Regenerating spent resin, destroying the perchlorate, and re-installing the regenerated resin is assumed to be an aggregate cost based on market forces. Regenerated resin has been demonstrated to maintain perchlorate capacity similar to virgin resin through three regeneration cycles in this demonstration and through at least 10 regeneration cycles in laboratory studies. Resin installation is often priced to include disposal. The cost of installing resin has been separated from the cost of incinerating spent resin. In conventional single-use IX applications, spent resin is typically incinerated at the end of its service life.

Cost elements to build and operate resin bed perchlorate treatment as IIX are summarized in Table 4. In building the cost model, engineering judgment and recent resin and disposal costs were used for a notional site. Resin beds are expected to be similar at different sites with a number of trains operating in parallel.

Table 4. Cost elements tracked during IIX demonstration (150-gpm scale)**.**

Cost Element	Data Tracked During Demonstration	Cost
System design	Personnel/Labor, Reimbursables, Subcontracted Labor/Deliverables	$44,193
Installation	Equipment, Personnel/Labor, Materials/Reimbursables, Subcontracted Services	$131,671
O&M	Personnel/Labor, Materials/Reimbursables, Lab Analysis, Other Subcontracted Services	$289,998
Regeneration of resin	Regeneration (includes shipping), Personnel/Labor, Materials/Reimbursables	$110,081
Destruction/waste disposal	Destruction of perchlorate residuals (two rounds), disposal of wastewater residuals	$36,432

7.2 COST DRIVERS

The cost of IX as a perchlorate treatment technology depends on many factors including the treatment flow rate, concentration of perchlorate in the water, and the concentrations of competing ions, predominantly nitrate and sulfate, in the water. The perchlorate and competing ion concentrations are critical to the service life of the resin. The design of the resin beds and the resin volume in each bed are expected to be similar across sites.

The primary factor affecting cost of IIX will be the price of resin regeneration and perchlorate destruction. Calgon, as the licensee of the regeneration and perchlorate destruction technologies, will likely negotiate market prices for the regeneration and perchlorate destruction. The two major competing market forces are the cost of virgin resin and the cost of incinerating spent resin. The cost of the technology must be considered in developing pricing for this service including capital, transportation, labor, reagents, and analytical costs.

7.3 COST ANALYSIS

To provide a basis of comparison for single-use IX and IIX, the life-cycle costs were estimated for perchlorate treatment at a single generalized facility. The generalized facility has a treatment capacity of 4000 gpm provided through two parallel IX vessel trains that each consist of two vessels configured in series. Each vessel contains 424 ft^3 of perchlorate-selective resin. Capital cost includes first virgin resin fill. When the lead bed of resin becomes saturated, the lead bed resin is removed and replaced with fresh resin, either virgin resin or regenerated resin. The lag bed becomes the lead bed and the bed with fresh resin (the former lead bed) becomes the lag bed. Operations are assumed to continue in this fashion for the life of the perchlorate treatment system, assumed to be 30 years. The capital cost associated with conventional single-use IX and with IIX are identical.

O&M costs are similarly assumed to be equivalent using single-use resin and IIX exclusive of the resin changeout. No increases in operations labor, laboratory, maintenance, electricity, or other direct costs were indicated based on demonstration experience. A minor savings associated with decreased water purging requirement of regenerated resin compared to virgin resin was ignored for the purposes of this cost analysis.

Based on this demonstration, 200,000 BVs are required to achieve perchlorate saturation of the IX lead bed in either conventional single-use IX or IIX application. No degradation of resin performance was indicated due to IIX during this demonstration or prior bench testing; an eight loading cycle service life was assumed for IIX due to potential physical or chemical degradation of resin, though none was apparent during this demonstration. Upon saturation of the lead bed, the lag bed will be effectively pre-loaded with (unremoved perchlorate from the lead bed) the equivalent of 50,000 BVs at the time of resin change-out. In conventional single-use application, resin removed from the lead bed is assumed incinerated. In IIX application, the resin removed from the lead bed is removed and regenerated in a regional facility and returned for re-use at the next service interval for a total of eight perchlorate loading cycles; after eight loading cycles, the resin is assumed to be incinerated. The notional system is expected to treat 2102 million gallons/year or 6452 acre-feet/year in either single-use IX or IIX application.

Cost of virgin resin was assumed to be \$246/ft^3 including sales tax. Regeneration is estimated at \$198/ft^3 on a contract basis to install regenerated resin inclusive of resin storage and disposal of residuals. Spent resin incineration is estimated at \$20/ft^3. Over 30 years of operation, 126 resin change-outs are anticipated resulting in 112 resin bed regenerations. The model costs for the perchlorate treatment system are summarized in Table 5. After 30 years of operation, the treatment facility is assumed to be scrapped at no net cost except the cost of incinerating the remaining resin. NPCs are calculated using a real interest rate of 2.7%. It is clear that IIX will result in cost savings as the price for regeneration service is less than the cost of purchasing virgin resin and incinerating the spent resin. Over 30 years of operation, single-use IX is predicted to cost \$100.16 per acre-feet compared to \$88.37 per acre-feet for IIX. IIX is modeled to save \$2,194,002 in NPC over a 30-year life cycle for the model facility.

Table 5. Perchlorate treatment system life-cycle costs.

Cost Element	Unit Cost	IIX NPC	Single Use Resin NPC
Treatment system design		$64,050	$64,050
Treatment system construction & commissioning		$1,942,997	$1,942,997
Treatment system operations & maintenance	$340,820/yr	$6,967,272	$6,967,272
Virgin resin	$246/ft^3	$992,140	$8,929,262
Spent resin regeneration	$198/ft^3	$6,388,415	$0
Spent resin incineration	$20/ft^3	$95,513	$740,808
Total NPC		**$16,450,387**	**$18,644,389**

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8.0 IMPLEMENTATION ISSUES

In general, systems for the treatment of drinking water are governed by the SDWA and their residuals by the Clean Water Act (CWA). Regulatory agencies generally require that materials or processes used for drinking water treatment be certified by the NSF. Calgon is pursuing NSF certification of the regeneration system. Residuals from regeneration and destruction operations are regulated under the Resource Conservation and Recovery Act. The destruction facility may also require Calgon to take steps for Clean Air Act compliance. Naturally occurring radionuclides can be accumulated on anion exchange resins; thus the requirements for technologically enhanced naturally occurring radioactive materials need to be considered. Additional permit and regulatory requirements that may apply in some jurisdictions include zoning and building permits for system installation.

The main end-user issues for this technology are cost, reliability, and availability. This technology is an extension of this currently available, widely applied technology. This technology can be added to existing perchlorate treatment systems without adding infrastructure. Implementation of this technology does not require a user to continue with this technology. Additional end user concerns may include:

- Costs for virgin resin include sales tax based on California rates. Sales tax may not be applicable to some end users and may be assessed at different rates depending on the location of the treatment site.
- The actual service life of regenerated resin is not absolutely known. A certification of exchange capacity and other physical properties of regenerated resin by the supplier may be required, similar to virgin resin.
- Halogenated VOCs appear to result from regeneration. Evidence suggests this is an artifact of regeneration system construction magnified by the small-scale of operations.
- Halogenated VOCs appear to also be formed in the perchlorate destruction reactor. Management of these VOCs will likely be eliminated in practice by using alternate perchlorate destruction techniques.

Resin regeneration—IIX—can be considered to be a “green technology” providing environmental benefits. For example, Calgon estimates that the carbon footprint for the regenerated resin is 12.5% that of single-use resin (Drewry, 2009). The perchlorate-selective IX resin demonstrated is sold by multiple vendors including Purolite and Thermax-USA. The regeneration process is also applicable to nitrate-selective resins used for perchlorate treatment and most perchlorate-selective resins (except Rohm and Haas PWA-2). The regeneration and perchlorate destruction processes demonstrated are currently being commercialized by only one source, Calgon, under the trade name CRS (Custom Resin Regeneration Service). The U.S. federal government can however use the technology without paying a license fee. Government entities may wish to use a sole-source justification process or to structure their requests for proposal broadly enough to allow the use of the IIX process.

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APPENDIX A

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